

# High Pressure Diffraction Studies of Zeolite Na-A (LTA)

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Abstract No. hrl9563

Beamline(s): X7A

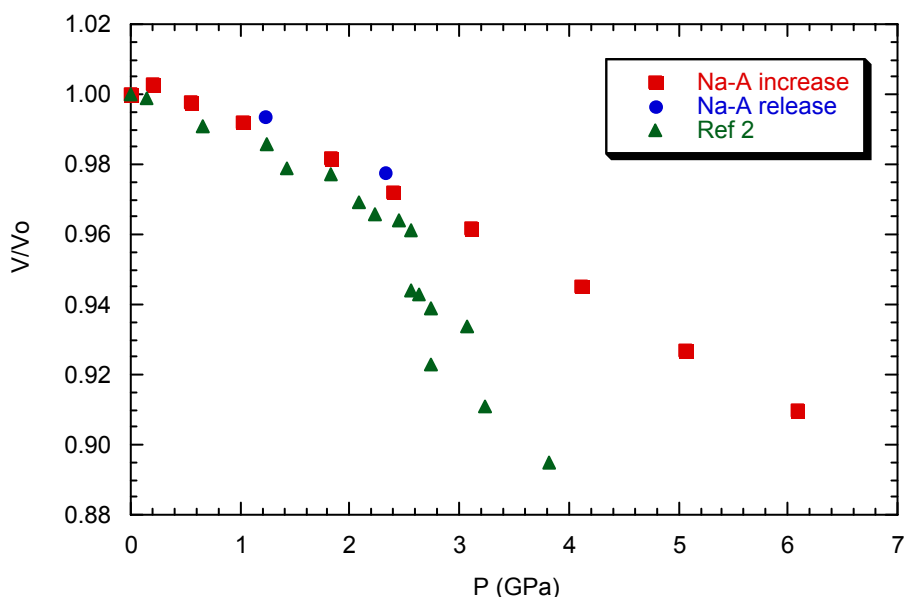
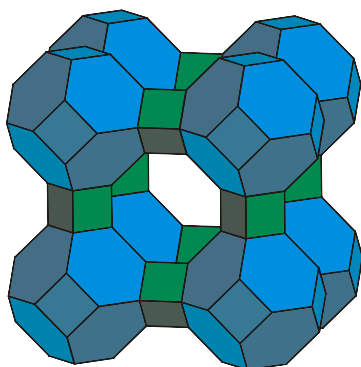
**Introduction:** Even though zeolites are synthesised under pressure using hydrothermal methods and often operate as catalysts under pressure, surprisingly few details of their behavior or structures under pressure have been reported. Zeolite Na-A (LTA) is perhaps the most prototypical of the zeolites, composed of a primitive cubic array of sodalite cages joined through double 4-rings, **Figure 1**. Previous *in-situ* diffraction studies of zeolite Na-A under applied pressure are limited to the work of Hazen *et al.*, [1,2]. These single crystal studies were undertaken in a diamond-anvil cell up to 4 GPa using six different hydrostatic pressure media: water, a 4:1 mixture of methanol and ethanol, methanol, ethanol, glycerol and the organofluorocarbon FC-75 ( $C_8F_{16}O$ ). One interesting observation in this work was that, when using the mixed methanol/ethanol medium, there were volume discontinuities at 2.0, 2.5 and 3.2 GPa. This was interpreted as a series of transitions between cubic phases, with volume changes of 0.5, 1.9 and 2.1% for transitions I  $\leftrightarrow$  II, II  $\leftrightarrow$  III and III  $\leftrightarrow$  IV, respectively, but no explanation of the structural changes was given. More recently, Secco and Huang interpreted the results of ionic conductivity studies of Na-A as evidence for an amorphisation at 4.6 GPa [3]. We have undertaken a series of studies aimed at further exploring this system and elucidating structural details.

**Methods and Materials:** A small amount of zeolite Na-A was loaded into a 200  $\mu m$  hole drilled into an Inconel gasket along with a few small ruby chips, soaked with a 16:3:1 mixture of methanol/ethanol/water and sealed in a modified Merrill-Basset diamond anvil cell. The pressure was determined based on the position of the R1 ruby emission line. The beamline was configured with an asymmetrically-cut Si(220) crystal bent to cylindrical curvature to provide horizontal focus, and a linear PSD. Diffraction data were collected from ca.  $2.5$ – $27.5^\circ$  at a wavelength of 0.6949 Å. Individual peaks were fit using a local least-squares program to pseudo-Voigt peak shapes to obtain accurate positions, and these were then used to determine accurate lattice constants.

**Results:** As shown in **Figure 2**, our data up to ca. 2.2 GPa matches that previously reported. Surprisingly, we see no evidence of the phase transitions previously reported by Hazen *et al.* at higher pressures. The reason for this is not clear, and further experiments are planned. Our results also contradict the report from Secco and Huang regarding an amorphisation, we see no evidence for this to the limits of our experiment.

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**References:** [1] R.M. Hazen, *Science*, **219**, 1065 (1983). [2] R.M. Hazen and L.W. Finger, *J. Appl. Phys.*, **56**, 1838 (1984). [3] R.A. Secco and Y. Huang, *J. Phys. Chem. Solids*, **60**, 999 (1999).



**Figure 1.** Structure of zeolite A.

**Figure 2.** Normalized volume vs pressure for zeolite Na-A